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**DURIP: Mechanical Testing System for MEMS and Small Scale Samples at
Cold and Elevated Temperatures**

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14. ABSTRACT A mechanical testing system was acquired by this DURIP grant for mechanical characterization of micron and millimeter-scale specimens at low (> -150 °C), and room to moderate temperatures (0-315 °C). The acquired instrumentation utilizes magneto-mechanical actuation that permits mechanical testing of micron and millimeter size specimens requiring ultra high force resolution (0.5 mN) and fine displacement control (~50 nm), which are not possible with conventional servohydraulic or DC motor based mechanical testing machines. The equipment has been integrated in the PI's laboratory and has already facilitated AFOSR supported research on fracture of silica epoxy nanocomposites. It is expected that it will further be of service in future AFOSR research on small-scale measurements in MEMS and thin films.		

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SUMMARY

A mechanical testing system was acquired by this DURIP grant for mechanical characterization of micron and millimeter-scale specimens at low ($> -150\text{ }^{\circ}\text{C}$), and room to moderate temperatures ($0\text{-}315\text{ }^{\circ}\text{C}$). The acquired instrumentation utilizes magneto-mechanical actuation that permits mechanical testing of micron and millimeter size specimens requiring ultra high force resolution (0.5 mN) and fine displacement control ($\sim 50\text{ nm}$), which are not possible with conventional servohydraulic or DC motor based mechanical testing machines. The equipment has been integrated in the PI's laboratory and has already facilitated AFOSR supported research on fracture of silica epoxy nanocomposites. It is expected that it will further be of service in future AFOSR research on small-scale measurements in MEMS and thin films.

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TECHNICAL DESCRIPTION

The acquired mechanical testing instrumentation by this DURIP grant provide invaluable infrastructure for the mechanical characterization of MEMS and small-scale specimens at low and moderate temperatures (-150 - 315°C). This instrumentation is a commercial mechanical testing system integrated with a low/high temperature chamber. The original plan for a high temperature furnace has been modified because of the sensitivity of the main instrument actuator to temperature, and the lack of proper thermal insulation without interfering with the instrument operation. The acquired instrumentation allows for measurement of deformations (elastic, yield, and ductility), failure (creep and fracture strength), fatigue (high, ~200Hz, and low cycle) and fracture properties (fracture toughness) of small-scale and millimeter-scale specimens. Integration of these capabilities is considerably more difficult compared to conventional servohydraulic testing machines because of the high sensitivity of the load measuring system to thermal loads and frictional forces. It is these capabilities that make the acquired instrumentation especially valuable to the PI's research.

This instrumentation has already been tested and used by the PI's student to measure the fracture toughness of millimeter-scale nanocomposite samples fabricated by the Air Force Research Laboratory (AFRL) at the Wright Patterson Air Force Base (WP-AFB). It is also planned to be used in proposed AFOSR research on fracture of thin films for MEMS and the effect of high temperature on the mechanical behavior of materials for MEMS. In the next sections the acquired instrumentation is presented and discussed.

I. Description of mechanical testing system by BOSE/EnduraTEC

The mechanical testing system (model: ElectroForce 3200, Figure 1) has been acquired from BOSE/EnduraTEC. It was installed on vibration isolation, air-suspended, optical table (Kinetic systems [1]) to reduce laboratory vibrations that limits the force resolution required for small samples, *e.g.* MEMS. The total cost of this part of the acquired instrumentation (incl. suspension tables) was approximately \$120,000. This ElectroForce 3200, apparatus can also be installed horizontally and be used in conjunction with an Atomic Force Microscope (AFM) at the PI's laboratory for room temperature nanoscale mechanical deformation measurements. According to BOSE/EnduraTEC data sheets [2] the peak-to-peak displacement range of the base system for 100 Hz modulation frequency is several hundreds of microns. In addition, BOSE/EnduraTEC integrated a high-resolution force transducer and load cell on the system, which make the mechanical testing apparatus very versatile: The same system can be used to test thin films with mN force resolution, or millimeter scale composite samples with 100-200 N ultimate force requirement.

In Table I, the base and the high resolution transducer system configurations are presented. The base system had been integrated with a cold/hot temperature chamber before, so this aspect of system integration was rather effortless. BOSE/EnduraTEC worked with us and implemented the low force/high resolution transducer available on their smaller systems (ElectroForce 3100) with the hot/cold chamber. The advantage of this combination is a wide range of testing capabilities with main advantages in the areas of thin films.

Table I. Specifications of BOSE/EnduraTEC ElectroForce 3200 mechanical testing system customized for high resolution, high temperature measurements. [2]

	Base configuration	High resolution
Max. dynamic or static force capacity	± 225 N (50 lb)	± 22 N (5 lb)
Min. controllable peak-to-peak force	6 mN (0.001 lb)	0.5 mN (50 mg or 0.00011 lb)
Min. controllable peak-to-peak displacement	0.0002 mm (0.00001 in)	0.00005 mm (0.000002 in)
Stroke	12.5 mm (0.5 in)	0.5 mm (0.02 in)
Min./max. frequency	0.00001 Hz - 400 Hz	0.00001 Hz - 200 Hz
Temperature Range	-150 – 315 °C via chamber	-150 – 315 °C via chamber
System life	> 15 billion cycles	> 15 billion cycles



Figure 1. Mechanical testing system (ElectroForce 3200) in vertical position integrated with the hot/cold chamber.

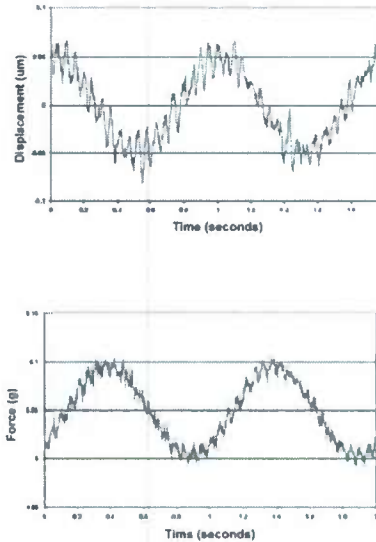


Figure 2. Displacement and force resolution benchmark tests, showing milligram forces and nanometer displacements.

The acquired system provides small forces and small displacements by a magnetic linear motor adopted from BOSE (parent company of EnduraTEC). This actuation allows for low force high frequency fatigue testing that is not possible with technologies developed for large specimen testing. The integrated electromagnetic actuator uses a flexure that moves in a frictionless manner to impose displacements. The 1000:1 ratio of the axial to lateral stiffness of this flexure results in very good lateral stability. The 20 nm ultimate resolution of electromagnetic actuation can be matched only by piezoelectric actuators. This flexural suspension allows for very small resolution in applied displacements and also high resolution in measured forces. Figure 2 shows the displacement and force profile for a fatigue-loaded specimen. These low force/displacement capabilities are unique in this transducer design. The need for low force and high cycle loading in thin film and MEMS type applications does not allow the use of servohydraulic or DC motor based mechanical testing machines.

This BOSE ElectroForce EF3200 system has been acquired with the following accessories that facilitate high/low temperature testing:

1.a. Low/high temperature chamber

The system configuration for measurements in the range (-150) - 325 °C is shown in Figure 3. The cold/hot temperature chamber allows for flow of inert gases but it does not support complete removal of air due to the clearance at the chamber top and bottom openings where the extension rods are connected to the specimen grips. MEMS samples are very fragile and the use of bellows is prohibitive due to the associated substantial frictional forces. BOSE/EnduraTEC provided software and hardware integration of this chamber. To test the system, we ran the chamber to 150 °C which is the T_g of epoxy-silica nanocomposites acquired from WP-AFB (see later discussion on fracture experiments with the same samples).



(a)



(b)

Figure 3. (a) Front and (b) rear side of hot/cold chamber integrated with the ElectroForce 3200 mechanical testing system.

Indeed, we found that the T_g of the nanocomposite samples vary with particle loading and the composite samples had lower T_g than the neat EPON epoxy.

I.b. High temperature measurement system for submicron samples, MEMS

The original plan called for the acquisition of a furnace for high temperature tests of MEMS materials. After significant deliberations with the BOSE/EnduraTEC the integration of high temperature furnace was abandoned because it would expose the actuator at the top of the instrument to temperatures higher than 110 °C, which is the maximum allowable temperature. Perforated extension rods would not reduce the thermal load to the actuator.



Figure 4. Microscope objective with 4× magnification for mid-wave thermal imager (IR). It will be used to measure temperature along miniature specimens that are heated resistively.

Because of the risk of damaging the instrument by integrating a furnace, we requested a no cost extension on this grant to explore other options for high temperature testing of miniature thin film samples. We decided to proceed with Joule heating of MEMS specimens accompanied with IR imaging. Because of the miniature size of MEMS specimens it is not accurate to place a thermocouple in their vicinity to measure temperature. Instead, a non-contact method was needed to acquire temperature data along their length. It was originally planned to acquire a pyrometer for direct measurement of temperature at a point on MEMS scale samples. Discussions with companies (Omega) pointed out to the potential inefficiency of these point-wise measurement systems (large spot size). Since an IR camera was available at our laboratory, a high magnification IR objective (4× which is the highest

magnification objective in the market) was acquired from Janos Technology (model: ASIO series, microscopic objectives), that is seen in Figure 4. The objective will be first mounted on an acquired bare bones microscope by Olympus and the IR camera we already have in the lab will be fixed onto the microscope frame. Image data will be streamed to a personal computer. The cost of this assembly was approximately \$27,000.

This is a custom-made series of objectives produced at this magnification only by Janos and with 4 months lead time. We finally received the objective assembly in March 2008, after ordering them in November of 2007. We will be testing this assembly in the next months. The objective in Figure 4 provides the maximum resolution that is practical for a mid-IR objective/camera system. This results to a field of view of approximately 4 mm. For a 256×256 pixel IR sensor, the resolved pixel size is 15 μm. Given that mid-IR wavelength is 5-8 μm, the resolution of this objective is the most we could expect for MEMS measurements. The largest IR sensor resolution that is meaningful for this objective is 512×512 pixels which can be borrowed from a central facility at UIUC.

Professor Chung-Seog Oh, who is an Associate Professor at Kumoh National Institute of Technology in Korea, will spend his sabbatical at the PI's laboratory in late 2008. Professor Oh has conducted high temperature MEMS experiments with MEMS before and he will start the

high temperature experiments with MEMS scale samples at the PI's laboratory. With the aid of the equipment that has been acquired by this DURIP grant, Prof. Oh will use this time significantly more advanced instrumentation.

II. Application of acquired system

Mode-I fracture tests of silica nanocomposites with 12-nm individually dispersed particles, fabricated at the WP-AFB, were carried out by the tabletop universal testing machine ElectroForce 3200, at a rate of 0.12 mm/min, Figure 5. The applied load was measured by a 50 lb loadcell (Honeywell Corporation). Figure 6(a) shows a typical load-displacement plot of one of these fracture experiments. In all fracture tests, the maximum load was less than 210 N, which corresponded to an elastic far-field stress of 10.5 MPa.

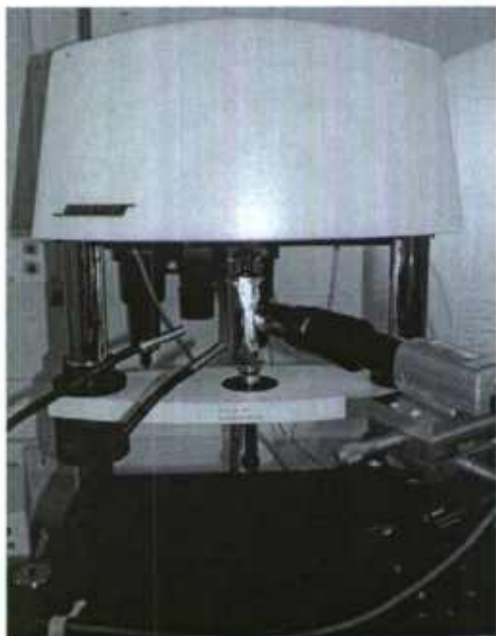
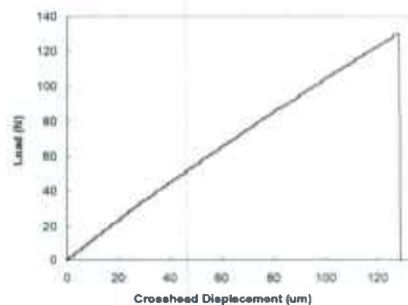
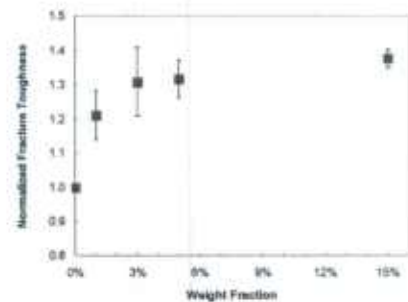


Figure 5. Fracture experiment with concurrent measurement of crack-tip field in a silica nanocomposite specimen [3] conducted with the acquired ElectroForce 3200 mechanical testing system.



(a)



(b)

Figure 6. (a) Load-displacement curve from a mode-I fracture test and (b), normalized toughness as a function of weight fraction of 12-nm silica nanocomposites. The data were collected with the instrument in Figure 5.

The normalized (by the toughness of the epoxy, 1.0 MPa \sqrt{m}) mode I composite fracture toughness was found to increase monotonically with silica weight fraction, Figure 6(b). Small weight fractions (1 wt.%) resulted in 20% increase in the effective toughness, but there was marginal difference in toughness between 3 wt.% and 5 wt.% composites. A 35% increase in toughness was measured for composites with 15 wt.% silica particles [3].

IV. Education and training of undergraduate and graduate students at UIUC

The acquired instrumentation will contribute to the educational mission of the PI and faculty at UIUC who teach and train graduate and undergraduate students in experimental mechanics. At the graduate level the students conducting their research in the aforementioned research fields will receive training in instrumentation that is the state-of-the-art in microscale mechanics of advanced materials, which will prepare them for their professional careers. At the undergraduate level, this instrumentation will support research by undergraduate students at the PI's lab. The PI has recruited eight undergraduate students in the past four years who conducted research in areas of high relevance to the PI's research program. These students were supported during the summer and academic year by NSF funds for Research Experience for Undergraduates (REU), and other summer undergraduate research programs sponsored by UIUC.

V. References

- [1] <http://www.kineticsystems.com/page.php/id/141>
- [2] <http://www.enduratec.com/products/specs/ELF3100Series.pdf>
- [3] Q. Chen, C. Chen, A. Roy, and I. Chasiotis, "Effective and Local Mechanical Behavior and Fracture of Silica Nanocomposites", submitted to *Composites Science and Technology*, (2008).